



# AEC-NASA TECH BRIEF



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## Sintering Characteristics and Properties of PuS and PuP Are Determined

Preparation procedures for PuS and PuP compounds have been developed and analyzed with respect to microstructure and end properties by O.L. Kruger and J.B. Moser of Argonne National Laboratory. Their report has been published in "Sintering Characteristics and Properties of PuS and PuP," *Journal of the American Ceramic Society*, Vol. 49, No. 12, pp. 661-667, December, 1966.

Ceramic materials, other than oxides, have become of increasing interest as reactor fuels in the past few years. Included in these ceramic materials are compounds of uranium and plutonium with Group VA and VIA nonmetals that have cubic crystal structures and high melting points. The paper describes the sintering characteristics and properties of two high-temperature compounds of plutonium, plutonium monosulfide and plutonium monophosphide, which have NaCl-type crystal structures. Property measurements were made on well-characterized high-purity materials.

The compounds of PuS and PuP were prepared by high-temperature homogenization of the product from the reaction of  $H_2S$  and  $PH_3$  gas with partly decomposed plutonium hydride. All operations on these materials, including material synthesis, specimen preparation, and property measurements, were carried out in glove boxes in an inert atmosphere of high-purity nitrogen gas.

Studies were conducted to determine the best method of sintering pellets to high densities with a minimum of oxygen contamination. The pellets, 0.7cm in diameter by 1.3cm long, were formed at  $21.1\text{kg/mm}^2$  and heated at  $100^\circ\text{C}$  intervals for 2 hours. The sintering studies were performed in a vacuum of  $10^{-5}$  torr or in flowing high-purity argon at 1.3 atm. Geometric densities and weight changes were measured after each

heat treatment. Combined oxygen and nitrogen contents of the sintered specimens usually were about 0.01wt% above amounts in the homogenized powders.

Studies of the sintering characteristics of these pellets, fired in vacuum and in an argon atmosphere, showed that these materials can be sintered to densities slightly greater than 90% of theoretical. The sintering process appears to be complex due to changes of mechanism with increasing temperature.

Weight loss data indicate that PuP has a higher effective vapor pressure than PuS, but under conditions of fuel irradiation (1 atm pressure of inert gas), PuP has a much lower rate of vaporization.

Microstructural examinations and lattice constant measurements verify that monosulfide and monophosphide can exist as substoichiometric compounds. Curvature of the sulfur-rich phase boundary toward greater sulfur solubility at high temperatures is indicated from X-ray data.

Density, melting point, thermal expansion, microhardness, Seebeck coefficient, electrical resistivity, thermal diffusivity, heat capacity, and thermal conductivity are also reported.

### Notes:

1. An evaluation of the sintering characteristics of PuS and PuP provides an understanding of the rate-controlling mechanisms involved in characterizing various polycrystalline materials. This information can enable industries using plutonium sulfides and plutonium phosphides to characterize their processing methods.
2. This information may be of interest to individuals or organizations involved in reactor materials, processing of ceramics, and the role of microstructures.

(continued overleaf)

3. Additional information is available in:

O.L. Kruger, J.B. Moser, "Preparation of the Sulphides and Phosphides of Plutonium," *J. Inorg. Nucl. Chem.*, Vol. 28, pp. 825-832, 1966. O.L. Kruger, "Preparation of PuC Pellets by Vacuum Sintering," *Nuclear Applications*, Vol. 1, pp. 348-355, August, 1965.

J.B. Moser, O.L. Kruger, "Heat Pulse Measurements on Uranium Compounds," *Journal of Nuclear Materials*, Vol. 17, pp. 153-158, 1965.

4. Inquiries concerning this report may be directed to:

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Source: O. L. Kruger and J. B. Moser  
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**Patent status:**

Inquiries about obtaining rights for commercial use of this innovation may be made to:

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